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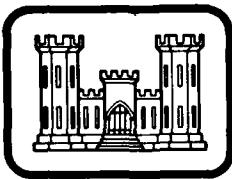
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REPORT NO. ONWI-249

STUDY OF A PROPRIETARY POZZOLANIC PRODUCT

by

Alan D. Buck

Structures Laboratory
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May 1981
Final Report

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Prepared for Office of Nuclear Waste Isolation
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Columbus, Ohio 43201

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) → Petrographic examination and limited chemical analysis were used to identify constituents in a proprietary "anti-corrosion" admixture (AD-644) for concrete. Petrographic examination included X-ray diffraction, light microscopy, and scanning electron microscopy. The CaO content of the sample was determined by chemical analysis as a basis for estimating cement content of the sample.		
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20. ABSTRACT (Continued)

The results indicated that the admixture was largely silica fume containing about 12 percent portland cement and a small amount of organic material which was believed to be a water-reducing admixture. Properties of a known silica fume (AD-536(2)) were used as a basis for concluding that silica fume was the principal constituent of this admixture (AD-644).
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Preface

The work described in this report was funded by the U. S. Department of Energy under Contract No. DE-AI97-81ET46633. The title of the investigation was "Investigation of Composition, Constitution, Properties, and Interactions of Materials Considered for Use in Plugging Boreholes and Shafts in Connection with Nuclear Waste Isolation." Mr. Floyd L. Burns, Office of Nuclear Waste Isolation, Battelle Memorial Institute, Columbus, Ohio, was Project Manager.

The work was done in the Structures Laboratory of the U. S. Army Engineer Waterways Experiment Station (WES) under the direction of Mrs. Katharine Mather. Messrs. Bryant Mather and John M. Scanlon, Jr., were Chief, Structures Laboratory, and Chief, Concrete Technology Division, respectively. This report was prepared by Mr. Jay E. Rhoderick.

COL N. P. Conover, CE, was Director of WES during this period. Mr. F. R. Brown was Technical Director.

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STUDY OF A PROPRIETARY POZZOLANIC PRODUCT

Samples

1. A 50-lb (22.68-kg) sample of AD-644 was received by the Structures Laboratory of the U. S. Army Engineer Waterways Experiment Station (WES) in December 1980.
2. Another sample of presumably similar nature had been examined for the WES Environmental Laboratory in February 1980. The results of that examination are presented in Appendix A.

Test Procedure

3. The sample reported on herein was examined by X-ray diffraction. A subsample of it was also examined with a stereomicroscope and as an immersion mount with a polarizing microscope. The sample was compared with a silica fume (AD-536(2)).
4. A 0.1-g sample of AD-644 was dispersed in 150 ml of ethyl alcohol for 3 hours in an ultrasonic cleaner. A few drops of the dispersed sample were placed on a sample stub, dried, coated, and examined with a scanning electron microscope (SEM).
5. A portion of the sample was put into solution with hydrochloric acid; this solution was analyzed by atomic absorption spectroscopy for calcium. By expressing the calcium as calcium oxide, assuming all of it was present in portland cement, and assuming a calcium oxide content of 63 percent for the cement, a cement content for the admixture was calculated.

Results

6. As indicated in Appendix A, the presumably similar material previously examined seemed to be a mixture of portland cement, an amorphous organic material, and amorphous mineral material that resembled silica fume. The X-ray pattern contained an amorphous halo like the

X-ray pattern of the silica fume AD-536(2), and the two materials looked alike in immersion mounts. Any glassy spheres of silica fume were too fine to be resolved at 630X with a polarizing microscope.

7. Since the presence of small spherical particles generally less than 1  $\mu\text{m}$  in diameter was shown by SEM examination, the presence of silica fume is considered proven. These spheres and a fragment of cement are shown in Figures 1 and 2. These spheres resemble those known to be in silica fume AD-536(2).

8. A value of 11.8 percent cement in the admixture was calculated by dividing 7.4 percent by 63 percent (assumed CaO content of cement). This is probably high enough to indicate cement was present as a deliberate addition rather than as a contaminant.

9. Since the supposed organic material resembled that found and identified in another admixture, it may be a high-range water reducer.

#### Summary

10. Examination of AD-644 indicates that it is probably a mixture of silica fume, about 12 percent portland cement, and an organic material.

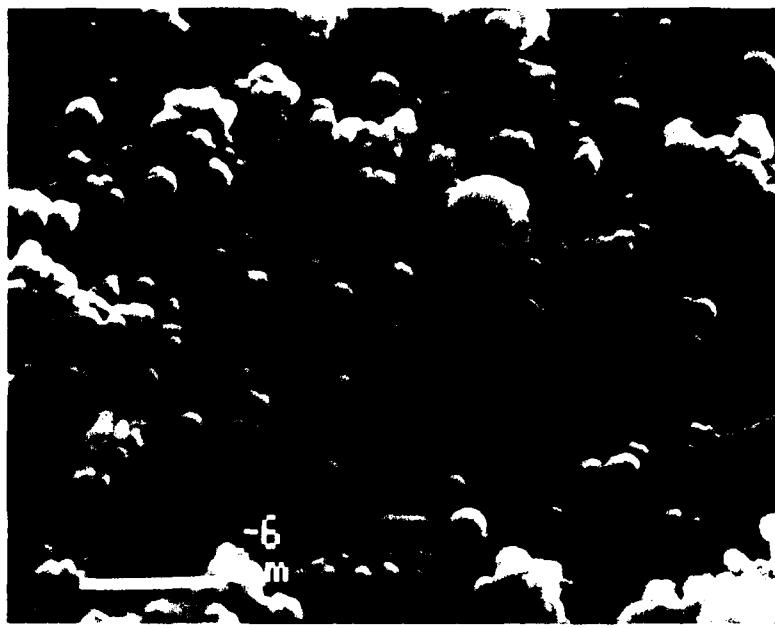


Figure 1. Micrograph 01 06 81-1, 20,000X, of dispersed AD-644. Spheres of silica fume can be seen.  
The reference bar is 1  $\mu\text{m}$  long



Figure 2. Micrograph 01 06 81-12, 5000X, of dispersed AD-644. Grain of portland cement adjacent to clusters of silica fume spheres. The reference bar is 10  $\mu\text{m}$  long

Appendix A: Results of February 1980 Laboratory Examination

WESSC

29 February 1980

MEMORANDUM FOR MR. PHIL MALONE, EL

SUBJECT: Limited Petrographic Examination of Admixture for Concrete

1. The material is said to make concrete resistant to acid attack. The sample was a fine gray powder. It was examined by X-ray diffraction and as an immersion mount with a polarizing microscope.
2. These examinations indicated that the sample is a combination of at least three different materials. These include:
  - a. Crystalline material that appears to be portland cement. No tricalcium aluminate appeared to be present in the cement.
  - b. Amorphous material that was too fine to be resolved by the light microscope. This material may be silica fume.
  - c. Larger amorphous spheres that are rapidly soluble in water. This is probably an organic substance.
3. Some of the sample was soluble in hydrochloric acid.
4. If additional characterization of the sample is needed, it is suggested that this include chemical analysis and examination with a scanning electron microscope. Physical tests of concrete containing this admixture could also be made to evaluate the claim that this material makes concrete acid-resistant.
5. In general, it should be pointed out that there are many such admixtures on the market and that most of them will not do what is claimed for them.

JAY E. RHODERICK  
Geologist  
Structures Laboratory

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14 January 1981

Errata Sheet

No. 1

STUDY OF A PROPRIETARY POZZOLANIC PRODUCT

Miscellaneous Paper SL-81-8

Report No. ONWI-249

May 1981

Page 1, Preface: Change the last sentence in the second paragraph to read

This report was prepared by Mr. Alan D. Buck.

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